

EXHIBIT D

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Applicant: Steve Say-Kyoun Ow and Tae Jin Eom

Serial No.: 09/121,152 Art Unit: 1731

Filed: July 22, 1998 Examiner: Anna Kinney

For: *BIOLOGICAL DEINKING METHOD*

Commissioner for Patents
P.O. Box 1450
Alexandria, VA 22313-1450

DECLARATION UNDER 37 C.F.R. § 1.132

Sir:

I, Howard Kaplan, hereby declare that:

1. I am employed at Enzymatic Deinking Technologies, Norcross, GA, as its chief operating officer. Enzymatic Deinking Technologies is the licensee of the above-identified patent application.

2. I instructed my laboratory manager, Jian Hua Ma, to conduct experiments to compare the deinking of recycled paper using the conditions described in Example 2 of Japanese Patent Application No. 59-9299 ("JP '299") and the above-identified application.

3. I reviewed JP '299 to determine the conditions and materials described therein for the enzyme enhanced deinking of recycled paper. The only conditions were described in the examples. Example 1 added a number of materials other than an enzyme and NaOH. Example 2 examined the effect of adding 1% by weight NaOH and an alkaline cellulase. It was my

understanding that the examiner preferred we use the conditions of Example 2 so that there would be fewer variables. We therefore conducted a comparison of the deinking of recycled paper as described in example 2, with the claimed method which requires a pH of less than 8, differing in the pH of the reaction mixtures and the cellulases which were added. Each experiment was performed 10 times to provide a statistically valid result. The results of the experiments are enclosed.

4. Example 2 of JP '299 does not provide a pH of the reaction mixture but instead refers to adding 1% (relative to the old newspaper) NaOH. The average pH of the mixture after caustic addition was 11.19. The average pH of the mixture after disintegration was 11.12. The average pH of the mixture after addition of the enzyme was 11.16 and the average pH of the mixture after stirring was 10.67. Each deinking example with 1% NaOH relative to wastepaper produces pHs in excess of 10.59. For purposes of comparison, NaOH was not added to the reaction mixture of the claimed method. The average pH of the reaction mixture after stirring was 7.5 and none were above 7.6.

5. It was not possible to obtain any of the enzymes described at page 3 of the JPA. We contacted Amano Pharmaceutical Co. and tried to locate Ueda Kagaku, listed as the manufacturers. We also searched a number of catalogs and on the internet. Amano did not sell the named enzyme and Ueda appears to be out of business. We then obtained an equivalent alkaline cellulase from Meiji Seika, HEP-100, an alkaline cellulase which is active over a range of at least 4.0 to 10.0, with a pH optimum of 8.0. For purposes of comparison, a neutral cellulase

was obtained from Novozymes, Novozym 342 produced by the fungus *Humicola insolens*, which has an optimum pH of between 6.5 and 7.5.

6. As described in Example 2 of JP '299, each reaction mixture contained old newspapers, cut in 2 x 5 cm pieces, fed into a laboratory disintegrator, water and, for the JP '299 study, 1.0% NaOH, relative to raw material old paper, and disintegration done at pulp concentration 5%, 40°C for 20 minutes. After disintegration, 0.2% enzyme relative to raw material old paper as described in example 2 was added to the mixture containing the 1% NaOH and an equivalent amount of enzyme added to the other reaction mixture, and stirring was done at 45°C for one hour. The pulped material was then concentrated to 15% pulp concentration, diluted to 1% by adding water, and filtered through a Buchner funnel. The paper in the funnel and the filtrate were then analyzed.

7. The whiteness of the treated pulp (L-value) and the whiteness of the removed liquid (L-value) were determined for paper and filtrates from both samples.

The results showed that the treatment at the lower pH was more effective than the treatment at the higher pH, in the presence of 1% NaOH. The whiteness of the claimed method was 2.2% higher than the JP '299 method. This increase in whiteness is material to deinking paper mills and would allow for less bleach consumption. It is also true that the measurement of the filtrate is higher with the claimed method, indicating more ink is removed with neutral enzymatic deinking.

	<u>Paper L-value</u>	<u>Filtrate L-value</u>
JPA sample with 1% NaOH	65.9%	60.0%
Ow sample at pH 7.2	68.1%	56.4%

8. Not only were the results superior without NaOH treatment, but the cost of the treatment in the absence of the NaOH is reduced since the price of NaOH, at the time the application was filed, was about \$400/ton. The absence of 1% NaOH in the claimed method would create a savings of approximately \$4.00/ton at the time the application was filed, or approximately \$6.80/ton today (See the attached abstract which discloses the price of caustic soda from 1988-1991). Mills typically process eight hundred tons per day, for a cost savings at the time the application was filed of \$3200/day, and operate 350 days/year year, leading to a cost savings of \$1.12 million/year as of the time this application was filed, or \$1.9 million today.

9. The undersigned declares that all statements made herein of his own knowledge are true and that all statements made on information and belief are believed to be true and further that these statements are made with the knowledge that willful false statements are punishable by fine or imprisonment or both under 18 U.S.C. 1001, and that such willful false statements may jeopardize the validity of the above-identified patent application or any patent issuing thereon.

Date: 2/22/06



Howard Kaplan

Effect of caustic and enzymes on pulp and filtrate whiteness with mixed ONP

12/6/2005

Exp. #	Caustic with alkaline enzyme										Standard Deviation
	1	2	3	4	5	6	7	8	9	10	
NaOH, %	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	
Alkaline MEU/HEP100, %	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2	
pH water	7.16	7.22	7.23	7.23	7.19	7.20	7.14	7.21	7.29	7.10	0.05
pH paper + water	7.18	7.17	7.16	7.20	7.16	7.23	7.18	7.24	7.29	7.17	0.04
pH after caustic addition	11.22	11.23	11.22	11.22	11.24	11.20	11.18	11.05	11.14	11.15	0.05
pH after disintegration	11.15	11.12	11.15	11.14	11.10	11.08	11.04	11.07	11.21	11.12	0.05
pH w/enzymes addition	11.14	11.19	11.18	11.24	11.20	11.13	11.15	11.04	11.08	11.22	0.05
pH final after stirring	10.70	10.67	10.70	10.89	10.85	10.89	10.89	10.82	10.76	10.87	0.05
Pulp whiteness, %	66.00	66.01	66.23	65.88	65.99	65.62	65.43	65.90	66.21	65.47	0.28
Filtrate whiteness, %	60.80	60.35	59.60	59.94	60.10	61.00	60.13	59.33	59.60	62.10	1.17
Enzyme only											
Exp. #	11	12	13	14	15	16	17	18	19	20	Average
Novozymes SP142, %	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2	Standard Deviation
pH water	7.16	7.18	7.16	7.20	7.17	7.16	7.18	7.16	7.16	7.16	7.17
pH paper + water	7.08	7.13	7.15	7.12	7.20	7.08	7.18	7.08	7.13	7.13	7.12
pH after disintegration	7.39	7.35	7.43	7.39	7.32	7.43	7.39	7.43	7.39	7.38	7.39
pH w/enzymes addition	7.39	7.38	7.43	7.39	7.32	7.42	7.40	7.43	7.39	7.39	7.39
pH final after stirring	7.51	7.47	7.55	7.51	7.44	7.55	7.51	7.55	7.51	7.50	7.51
Pulp whiteness, %	69.10	67.73	68.43	67.95	69.12	67.69	68.03	67.76	68.34	68.13	69.05
Filtrate whiteness, %	57.46	56.90	55.08	54.84	58.52	57.23	56.23	55.76	56.72	54.75	60.35

Procedures:

This set of tests was conducted following the procedure in the Example 2 in JP-A 56-929.

Mixed ONP was shredded into 2x5 cm pieces, 100 g was fed into a laboratory disintegrator with water and sodium hydroxide, the disintegration was done at about 5.0%, 40C for 20 minutes. After disintegration, 0.2% based OD fiber enzymes was added, and stirring was done at 46C for 1 hour. It was then concentrated to about 15% pulp consistency, diluted to 1% by adding water, and pulp sheet was made in a Buchner funnel. When the pulp was concentrated, the removed liquid was kept at 5C for 12 hours, and 200 ml of supernatant was taken out and the L value was measured.

Comments:

The tests were performed with mixed ONP collected around Metro Atlanta area in December 2005, which has high inherent whiteness than Asian ONP. The results from these tests shall be different from all previous tests in terms of whiteness gain and ink removal before and after treatment, however the relationship between with and without caustic, or at different pH shall be very similar.

1.0% NaOH shifted the pH from 7.1-7.2 to 11.2, and the final pH dropped slightly with NaOH due to fiber absorption of alkalinity, and the final pH increased without NaOH due to chemicals leaching from paperfibers to the suspension. Enzymes showed no impact on pH of the fiber suspension.

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Chlor-alkali prices: caustic rollercoaster.

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Since chlorine and caustic soda are made together in nearly equal amounts, their prices traditionally waxed and waned in opposing cycles. In general, when caustic soda was in demand, its price rose, but the extra chlorine produced was not wanted, so its price fell, and vice versa. However, the constancy of caustic prices in 1987 was believed to mark a fundamental change in this cycle and the greater demand for caustic was expected to control prices more than chlorine from then onwards (chlorine has fallen out of favour on environmental grounds). The theory has been thrown into confusion by the fact that caustic prices have dropped sharply over the past 2 years. Spot prices for caustic soda in Western Europe have fallen to around \$ 50/tonne from about \$ 400/tonne in 1988-1991. Chlorine demand in Western Europe has also fallen from a peak of 10,000 tonnes/y in 1989 to only 8500 tonnes/y now. The fall in demand for caustic soda has been attributed to reduced inter-regional deep-sea trade and to the fall in demand for caustic from alumina producers. Spot prices for caustic soda are expected to move sharply upwards in 1995 when the alumina market has settled down and to continue to rise up to the year 2000.

descriptor-chemical businesses generally; trends - general
general industrial code-MS-00; TR-40

cas substance name-chlorine
sic code-2812
cas registry number-7782-50-5
country-Western Europe
country code-11000
business term-market
fact date-1989-1994

cas substance name-caustic soda
sic code-2812
cas registry number-1310-73-2
country-Western Europe
country code-11000
business term-pricing
fact date-1988-1994

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